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EFFECT OF HEAT TREATMENT ON Al2024 REINFORCEMENT WITH MULTIWALLED CARBON NANOTUBE

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ABSTRACT

In this research paper, Multiwalled carbon nanotubes (MWCNTs) reinforced with Al2024 were ball milled for homogenization of powder. Nanocomposite was developed using MWCNT reinforcement having 0.5%, 1.5%, and 2.5% weight percentage mixed with Al2024 by powder metallurgy technique. Samples were compacted using a hydraulic press and sintered. Further, sintered samples were solutionized by heating at 560º C followed by quenching and ageing, to explore the enhancement of properties. Experimental findings reveal that there is an improvement in micro hardness of the composite from 27 HV to 45 HV with increasing in hardness of 66.66% in case of a sintered composite, whereas tensile strength increases from 81.312 MPa to 131 MPa indicating the increase in strength up to 61.09%. Sintered-heat treated composites showed the improvement in micro hardness of 31 HV to 56 HV. Additional improvement of hardness and tensile strength recorded was 13.98% and 17.78% respectively for sintered-heat treated composites. Density decreases with an increase in the addition of MWCNT and porosity increases with increase in percentage reinforcement for both the sintered and sintered-heat treated composites. SEM and XRD images exhibited the presence of uniformly dispersed MWCNT with the Al2024 matrix.TEM images revealed that composite strength has been increased due to the entangled unbroken clusters of the nanotubes.

KEYWORDS: Al2024 Alloy, Multiwalled Carbon Nanotubes, PM Technique, Sintering, Heat Treatment & Mechanical Properties

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Original Article

INTRODUCTION

Multiwalled carbon nanotube (MWCNT) is a low density, high strength and hardness material together having good mechanical, thermal and electrical properties. Several researchers have used the MWCNT to enhance the properties by reinforcing with different metallic materials. MWCNT is promising material, because of its mechanical strength (15-20 times more than steel), density (five times lighter than steel), and thermal conductivity almost five times that of copper [1]. MWCNTs composites are promising materials in light-weight automotive application, nonlinear optics, batteries, nano wires, sensors and high strength devices [2]. The hardness of Al +5% SWCNT exhibited 78% higher than that of Aluminium. An addition of 1% MWCNT to Aluminium, had shown enormous enhancement in yield strength (almost 500%) [3]. Further, nanomechanics of CNT composite indicated that SWCNTs have Young's modulus around 1TPa and can withstand 5-10% axial strength corresponding to the

stress of about 50GPa [4,5].

For instance, due to its enhanced physical, thermal and electrical conductivity, CNTs excel widely in industrial applications where the conventional material fails to exhibit. The work on the hot pressing technique of Al-CNT composites by others recorded that moderate increases in electrical conductivity with an increased volume fraction of the CNTs [6]. However, controlled mechanical milling of Al-CNT showed a strong interfacial bonding between matrix and reinforcement [7] exhibiting the yield strength of 620MPa with 4.5 Vol% of MWCNT and uniform dispersion of MWCNT.

Studies have shown that has improved the mechanical properties compared to the matrix. The data on the development and properties of MWCNTs composite using powder metallurgy route was carried out by few researchers [8] is scarce. Therefore, this work has been focused to develop MWCNT reinforcing with Al2024 light weight, high strength alloy for engineering applications.

EXPERIMENTAL DETAILS

Materials

Matrix material used in this work is Al2024 alloy having composition listed in Table 1. Various properties Al2024 alloy are presented in Table 2.

Table 1: Chemical Composition of Al2024 Alloy

Elements	Mg	Si	Fe	Cu	Zn	Ti	Mn	Cr	Al
Amount of Wt. %	1.5	0.5	0.5	4.5	0.25	0.15	0.5	0.1	Balance

Table 2: Properties of Al2024 Alloy

Parameters	Particle size, µm	Density, g/cc	Melting Point, º C	Brinell Hardness	Ultimate Tensile Strength, MPa	Ductility/ Elongation, %
values	70-75	2.78	638	120	469	19

Pristine MWCNTs powder was procured from NANOSHEL USA having 98% purity. Parameters of MWCNT used as reinforcing material are; 10-20nm x 2-6nm x 10-20µm size, the melting point is 3652-3697º C, density-1-2 g/cc.

METHODS

Composite Fabrication

Matrix material used in this work is Al2024 alloy was prepared by mixing each element using planetary ball mill at 200 rpm for a duration of 3h. Reinforcing material MWCNT’s powder was initially purified and then mixed with a concentrated Nitric acid (HNO_3). The mixture was filtered and washed with deionized water followed by drying at 120°C. Impurities such as graphitic particles, amorphous carbon or any other impurities present in MWCNT’s were removed during the process. MWCNT’s powder of 0.5%, 1.5%, and 2.5% weight percentage was mixed with Al2024 alloy powder. Matrix and reinforcing materials were again mixed in a planetary ball mill for a duration of 3h at 200 rpm [9,10,11]. Further, the mixture of each proportion was pressed in a hydraulic press of 1000 kN capacity [12]. A 180 kN load was applied on the mixed powder to achieve the final size of 20mm Dia x 55 mm length billets. These billets were sintered in a vacuum tube furnace about 2h at 580ºC in a nitrogen environment followed by air cooling [13].

Thermal Loading

Thermal loading of the sintered samples of both matrix and its composites were carried out in a muffle furnace having a heating temperature range of 20º C to 1000º C. Samples were first solutionized by heating to 560°C and holding at this temperature about 8h. Further samples were quenched in boiled water. Then artificial aging was carried out for 4h at 180°C.

Preparation of Samples for Density, Hardness and Tensile Tests

The Experimental density of the composite was determined using Archimedes principle for both the matrix and composites. Theoretical densities were calculated using the rule of mixture for the sintered and sintered-heat treated samples. Hardness tests and tensile tests were carried out according to ASTM E92 standard [14] and the ASTM E8 standard for both types of samples [15].

Morphological Study Using SEM, TEM and XRD

Morphological study of the Al2024+MWCNT composite was obtained using Scanning electron microscope and Transmission electron microscope. Scanning electron microscope (SEM-TESCAN VEGA3 LMU, Czech Republic) was used for microstructural analysis. TEM images of the Al2024+MWCNT composite prepared were taken from JEOL JEM-2100 LaB6 transmission electron magnifying instrument (TEM) operates at an accelerating voltage of 200 kV and a lattice resolution of 0.14 nm and magnification of 2 kX-1500 kX. Crystal structures of the powder and composite were analyzed by X-ray diffractometer (PANalytical, Netherlands) with Cu-Ka radiation.

RESULTS AND DISCUSSIONS

Measurement of Density

The experimental density of the composites was studied using the rule of the mixture and Archimedes principle [16] for different weight percentage of MWCNT (0.5%, 1.5%, and 2.5%) are shown in Figure 1. Density decreases with increase in MWCNTs addition may be due to the displacement of aluminium atoms by the light-weight carbon atom [17]. Further, the decrease in the density was attributed to increasing pores observed in sintered nanocomposite because MWCNT was processed in an HNO_3 environment in which carboxyl (–COOH) or hydroxyl (–OH) functional group exist. At sintering temperatures and heat treated composites of different reinforcing MWCNT, the impurities in the composite material were pushed out completely [18]. This leads to shrinking of the particles and reduction in the volume thereby enhancing the density of a given volume of Al2024+MWCNT.The experimental density of sintered-heat treated samples increases by 1.326±0.2388% compared to sintered composites. As the percentage addition of MWCNT increases, the density individually decreases to 1.11%, 1.49% and 1.52% in sintered and sintered-heat treated Al2024+MWCNT composites respectively.

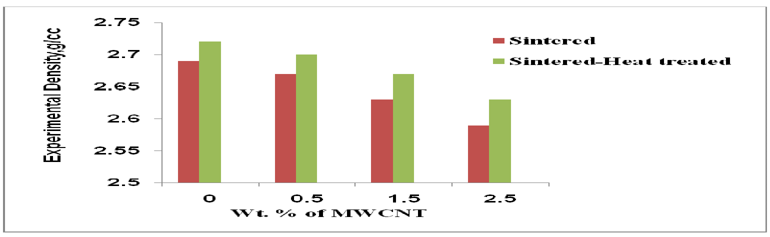

Figure 1: Experimental Density of the Composites with Varying Weight Percentage of MWCNTs

Measurement of Porosity

The porosity of Al2024+MWCNT sintered composites increases with increasing percentage of 0.5% to 2.5% MWCNT as presented in Figure 2. The porosity of the nanocomposite should be less for industrial applications. The percentage of porosity in the sintered composite is ranging from 3.27-6.159% for the varied weight percentage of MWCNT [19]. The decrease of porosity was observed to an extent of 16.65% to 8.71% in sintered to sintered-heat treated composite may be due to the dislocation movement of atoms and extension of grain boundaries by the addition of carbon nanotubes at high temperature during heat treatment [20]. The further reinforcement of MWCNT decreases the porosity marginally because its interfacial bonding hinders the movement during heat treatment of the nanocomposite.

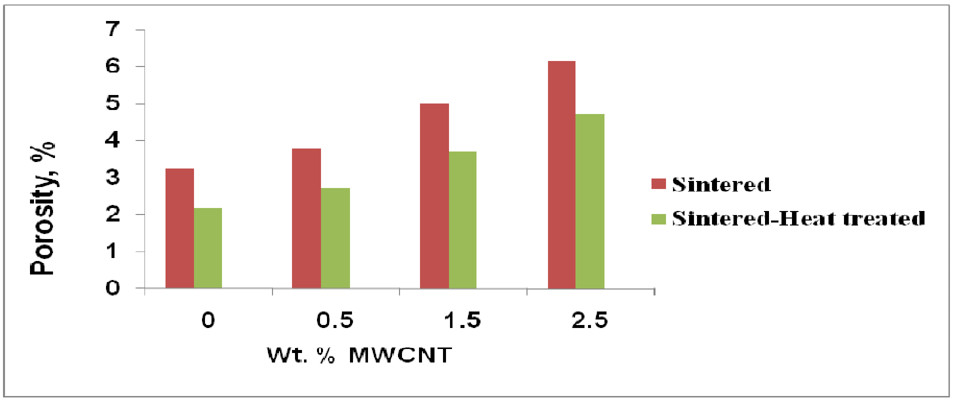

Figure 2: Porosity of the Composites with Varying Weight Percentage of MWCNTs

Micro Hardness Test

Hardness test was carried out using a Vickers micro-hardness tester is shown in Figure 3 for sintered and sintered-heat treated samples of the Al2024+MWCNT composites. Maximum hardness of 27HV was achieved for sintered Al2024 alloy and 45 Hv for 2.5% MWCNT sintered samples indicates that addition of MWCNT dispersion is uniform and observed at the interfacial space [21]. Further, sintered-heat treated composites recorded 56Hv exhibiting an overall increase of 24.8%. The increase in the hardness may be attributed to the formation of Al_4C_3 during heat treatment [22]. The uniform interfacial bonding between matrix and reinforcement was considered as prominent factor for the increase in the hardness [23,24,25]

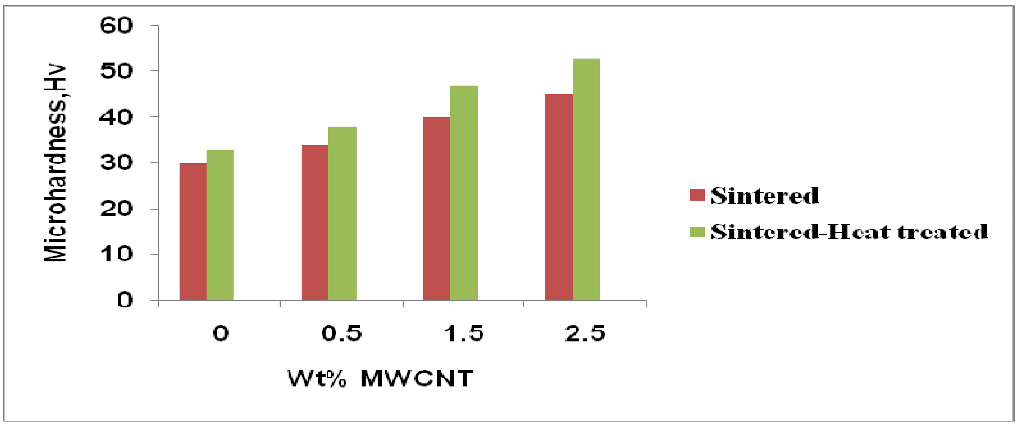

Figure 3: Microhardness of of Sintered and Sintered-Heat Treated Nanocomposite Plotted Against Wt% of MWCNT

Tensile Test

Ultimate tensile strength (UTS) of the composites (Figure 4) showed a significant increase in the tensile strength in case of sintered-heat treated composites when compared to sintered composites [26]. The increase of UTS from 81.32 MPa to 131 MPa in case of sintered composites may be due to the addition of 0.5%-2.5%MWCNT, which is about 61.02%. In the case of a sintered-heat treated composite, maximum of 168MPa (2.5%MWCNT) was achieved from 93.92MPa (0%MWCNT) [27]. An average increase of 17.78±3.16% has been achieved by the addition of MWCNT due to the heat treatment of nanocomposite [28, 29]. This phenomenon is due to interfacial bonding and entangled networks of bent nanotubes [30]. Heat treating of sintered nanocomposite enhances the UTS due to the mobility of large mass of atoms resulting in smaller grain sizes [31,32] due to applied tensile force. Increase in temperature during heat treatment fills the pores and reduces the imperfection which was observed during the sintering process. This is evident from the earlier research studies, the tensile load exerted on the composite transferred to the carbon nanotube thereby pulling leads to bending in nanotubes and break in the form of telescopic sheath[33,34]. The effective dispersion and better interfacial bonding between the matrix and the varied fraction of MWCNTs enhanced the UTS as reported by the others [35,36]. Thermal treatment is also a factor for enhancing the strength of Al2024+MWCNT composites

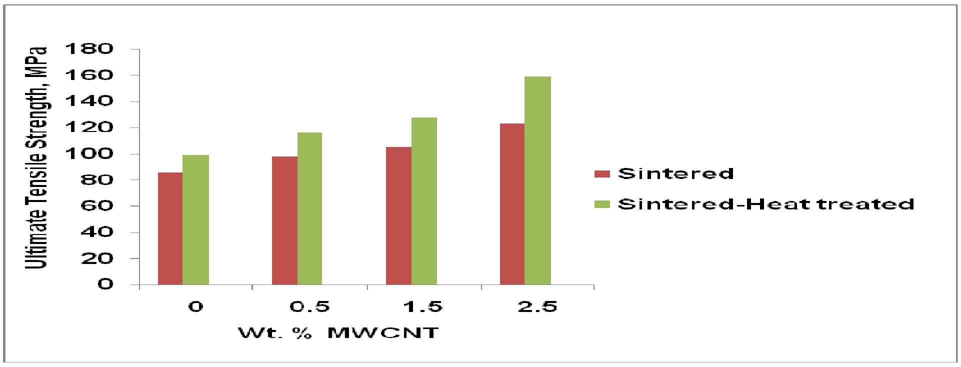

Figure 4: Ultimate Tensile Strength of Sintered and Sintered-Heat Treated Samples Plotted Against Wt% of MWCNT

Surface Morphology

XRD scans of Al2024+MWCNT are shown in Figure 5 and they show that presence of CNT is at 2θ=23.6º and 44.6º whereas high peaks of aluminium are present at uniform dispersed positions.

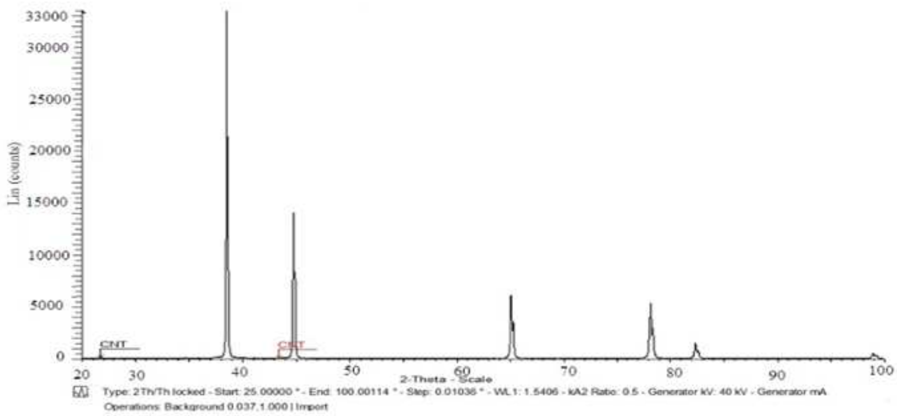

Figure.5: Enlarged XRD Image of Al2024+0.5%MWCNT Composite

Figure 6 shows the SEM image of Al2024+1.5%MWCNT powder after ball milling. Aluminium particles visible are of 200 mesh size was chosen for better compactness during sintering. TEM image shown in Figure 7 reveals the presence of entangled networks of carbon nanotubes in the Al2024+MWCNT nanocomposite. The distribution of MWCNT in the composite with bent nanotube appears in all the reinforced fractions of MWCNT are due to the higher rate of ball milling in order to achieve uniform dispersion of MWCNT. However, agglomeration MWCNT in the composite was reduced by controlling the duration of ball milling time not exceeding 10h.

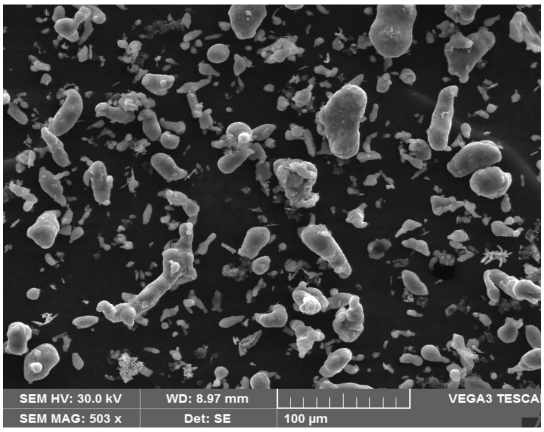

Figure 6: SEM Image of Al2024 Powder

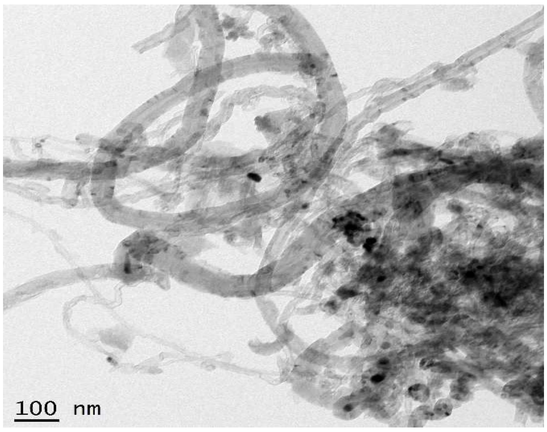

Figure 7: TEM Image of Al2024+MWCNT Nano composite

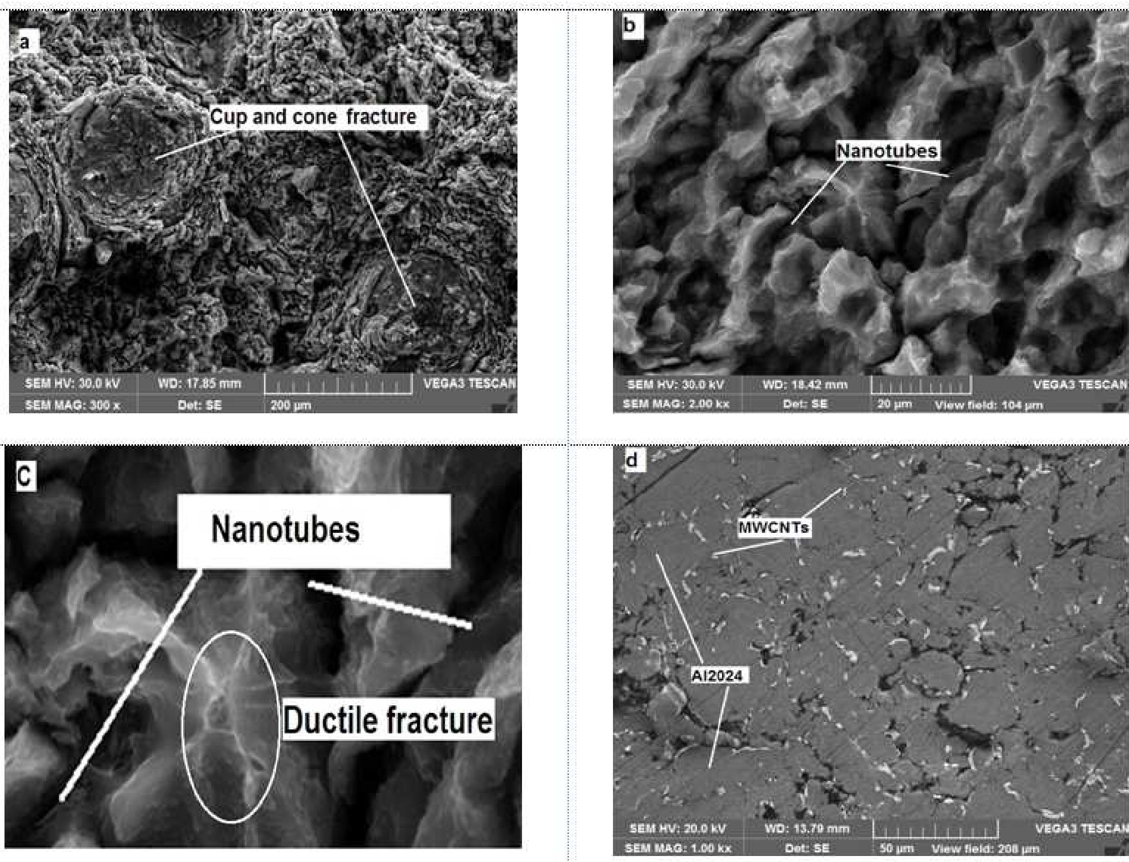

Figure 8: SEM Image of Al2024+1.5%MWCNT a) Tensile Fracture with Cup-Cone Failure b) Sintered-Heat Treated e Surface with MWCNT Fracture c)Enlarged Image of the X d) Surface of the Sintered-Heat Treated Surface with Distribution of Nanotubes at the Grain Boundary

SEM profile of sintered-heat treated composite shown in Figure 8a-8d represents the Al2024+MWCNT nanocomposites nature of fracture which is similar to the base aluminum fracture [37]. The presence of CNTs, Al-Cu and Al4C3 are observed after aging [33] may be due to the presence of elements present in the alloy and the reactions happened during heat treatment. Fracture micrographs of the Al2024+MWCNT composite displayed the resistance nature of nanotubes (Figure 8c) by way of deformation. The breaking of the material for the applied force due to which matrix pulls out and nanotubes breaks in telescopic sheath form as reported by Bakshi et al [35]. Bent nanotubes promote increase young's modulus during applied tensile load and are rigid in nature [30].

CONCLUSIONS

- Al2024+MWCNT nanocomposite with uniform dispersion was achieved by ball milling and fabrication was done using powder metallurgy technique.
- Sintered and sintered-heat treated composites exhibited presence of Al_4C_3 and enhanced the hardness with good interfacial bonding between matrix and reinforcement
- Strengthening effect is achieved by the bent nanotubes as observed during the sintering and sintered-heat treatment.
- SEM revealed a uniform distribution of CNT and TEM revealed the direction of the nanotube in the matrix for both sintered and sintered-heat treated nanocomposite
- Fractured surface exhibited the ductile fracture with a telescopic sheath like pulling of the nanotubes in the composite

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